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Methods for chemical analysis of iron, steel and alloy - The flame atomic absorption spectrophotometric method for the determination of nickel content

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Methods for chemical analysis of iron, steel and alloy - The flame atomic absorption spectrophotometric method for the determination of copper content

This standard applies to the determination of the amount of nickel in cast iron, carbon steel, and low alloy steel. Measuring range is 0. 005% ~ 0. 50%.

This standard follows the requirements GB 1467-78 “Method for chemical analysis of metallurgy product - General rules and regulations”.

1 Method summary

The sample is decomposed with appropriate mixed acid, perchloric acid is added to evaporate it to produce smoke, AND water is used to dissolve the salt. The sample solution is sprayed into the air-acetylene flame, and the copper hollow cathode lamp is used as the light source. The atomic absorption spectrophotometer is used to measure the absorbance at the wavelength of 232.0nm.

To eliminate matrix effects, when drawing the calibration curve, it shall add the iron the amount of which is similar to that of the sample solution.

2 Reagent

2.1 Perchloric acid (ρ1.67g/ml).

2.2 Hydrochloric acid-nitric acid mixture: MIX three parts of hydrochloric acid (ρ1.19g/ml), one part of nitric acid (ρ1.42g/ml), and two parts of water together; PREPARE it before use.

2.3 Nitric acid-perchloric acid mixture: MIX 100ml of nitric acid (ρ1.42g/ml) with 800 ml of perchloric acid (2.1); USE water to dilute it to 1l; MIX it uniformly.

2.4 Pure iron solution: WEIGH 10g of pure iron (nickel content less than 0.0005%); PLACE it into a 800ml beaker; ADD 100ml of hydrochloric acid-nitric acid mixture (2.2); HEAT to fully dissolve it; ADD 120ml of perchloric acid (2.1); EVAPORATE it until the perchloric acid produces white smoke; KEEP 1min; COOL it down; ADD 100ml of water; HEAT to dissolve the salts; after cooling it

down, TRANSFER it into a 250ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly.

2.5 Nickel standard solution

2.5.1 WEIGH 1.0000g of metallic nickel (<99.9%); PLACE it into a 250ml beaker; ADD 50ml of nitric acid (1 + 1); HEAT to dissolve it; COOL it to room temperature; TRANSFER it into a 1000ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. AND 1ml of this solution contains 1.0mg of nickel.

2.5.2 PIPETTE 10.00ml of nickel standard solution (2.5.1); PLACE it into a 100ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. AND 1ml of this solution contains 100μg of nickel.

3 Instruments

Atomic absorption spectrophotometer, equipped with air-acetylene burner and copper hollow cathode lamp. Air and acetylene shall be pure enough (containing no water, oil, or copper) to provide a stable and clear lean flame.

The atomic absorption spectrophotometer used shall reach the following indicators.

3.1 The minimum precision requirements: The calibration solution of the highest concentration is used for absorbance measurements for 10 times, AND the standard deviation shall not exceed 1.0% of the average absorbance.

The calibration solution of the smallest concentration (not zero calibration solution) is used for absorbance measurements for 10 times, AND its standard deviation shall not exceed 0.5% of the average absorbance of the calibration solution of the highest concentration.

3.2 Characteristic concentration: The characteristic concentration of nickel in a solution having a similar matrix with that of the final measurement sample solution shall be less than 0.10μg of nickel/ml (at wavelength 232.0nm).

3.3 Detection limit: The detection limit of nickel in a solution having a similar matrix with that of the final measurement sample solution shall be less than 0.15μg of nickel/ml (at wavelength 232.0nm).

3.4 Linearity of calibration curves: The ratio of the slope of the upper 20% concentration range of the calibration curve (expressed as the change in absorbance) to the slope of the lower 20% concentration range shall be not less than 0.7.

In eight 100ml volumetric flasks, respectively ADD 25ml of pure iron solution (2.4), then respectively ADD 0.00, 0.50, 1.00, 2.00, 4.00, 6.00, 8.00, 10.00 nickel standard solution (2.5.1); USE water to dilute it to the mark; MIX it uniformly.

4.4.2 Nickel content 0.10% ~ 0.50%

In seven 100ml volumetric flasks, respectively ADD 5ml of pure iron solution (2.4), then respectively ADD 0.00, 1.00, 2.00, 4.00, 6.00, 8.00, 10.00 nickel standard solution (2.5.1); USE water to dilute it to the mark; MIX it uniformly.

4.4.3 At the atomic absorption spectrophotometer at a wavelength of 232.0 nm, USE the air-acetylene flame; USE water to adjust zero; MEASURE the absorbance of the solution (4.4.1) or (4.4.2).

The absorbance of each solution of the calibration curve series minus the absorbance of the zero concentration solution is the net absorbance of the nickel calibration curve series solutions. The nickel concentration is used as the abscissa AND the net absorbance as the ordinate, to draw the calibration curve.

5 Calculation of analysis results

CALCULATE the percentage of nickel by the following formula:

$$N_i(\%) = \frac{(c_2 - c_1) fV}{m_0 \times 10^6} \times 100$$

Where:

c_2 - Concentration of nickel in the sample solution as found from the calibration curve, μg/ml;

c_1 - Concentration of nickel in the accompanied sample blank solution as found from the calibration curve, μg/ml;

f - Dilution factor, f = 1 when the nickel content of the sample is less than or equal to 0.10%; AND f = 5 if the nickel content of the sample is more than 0.10%;

V - Volume of the test sample solution, ml;

m_0 - Sample amount, g.

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